Audit of Data Quality
January 2010 Sampling Event
Data associated with "Ground Water Investigation in Pavillion, Wyoming," QA ID #G-14478
analyzed at US EPA Region VIII Laboratory in Golden, Colorado

ADQ Report Date: August 1, 2011
Four validation Excel spreadsheets are included in this ADQ report and are provided as separate files: January 2010 Pavillion R8 Volatiles Method 8260 Validation Worksheets, January 2010 Pavillion R8 Semivolatiles Method 8270 Validation Worksheets, January 2010 Pavillion R8 TPH DRO Method 8015D Validation Worksheets, and January 2010 Pavillion R8 TPH GRO Method 8015D Validation Worksheets. These worksheets include documentation of the validation process, along with sample and batch information, and recalculations.

1. Laboratory Data Audited:

Laboratory (Organization): US EPA Region VIII Laboratory.

Sample Type(s)/Methods/Analyte(s): Four analytical methods were included in this task: 1) TPH/DRO (EPA Method 8015), 2) Semivolatiles (EPA Method 8270), 3) Volatiles (EPA Method 8260) and 4) TPH/GRO EPA Method 8015).

Sample Identification: PGDW5, PGDW20, PGDW30 and PGDW32.
On January 22, 2010 twenty-two water samples were received under WO 1001002. On January 25, 2010 thirty-six waters, 9 soils and 1 holding water for VOAs was received under WO 1001003. Also on January 25, 2010, 4 waters and 1 RO filter were received under WO 1001005. WOs associated with these four samples are identified in the support Excel Spreadsheets provided with this Audit of Data Quality Report.

QA Reviewers: Rebecca Shircliff and David Gratson, Neptune and Company, Inc.

Method Information:
1) TPH/DRO: EPA Method 8015D (modified), Region VIII Operating Procedure (OP) ORGM-508 r1.0
2) 8270 semivolatiles: EPA Method 8270D (modified), Region VIII OP ORGM-515 r1.1
3) 8260 volatiles: EPA Method 8260, Region VIII OP ORGM-501 Rev 1.1.
4) TPH/GRO: EPA Method 8015D (modified) Purge and Trap, Region VIII OP ORGM-506 r1.0.

File Information: Final Report included in file 1001002, 1001003, 1001005 amend 2 09 jun 11 1029_S.pdf. Spreadsheet (EDD) containing results in file 1001002,1001003,1001005 AMEND 2 09 jun 11 1029.xls

TPH/DRO: Associated files: TPH/DRO Water Samples (WOs 1001002 [22 samples] & 1001003 [13 samples]).pdf. Case comments indicate 22 water samples were received on 1/22/2010 with thirty-six water samples, nine soil/sediment samples, one RO filter, and four precipitation samples plus QC were received on 1/25/2010 for the Pavillion project. Samples prepared and analyzed according to LSR 1001-004. For TPH/DRO specifically: Extraction using EPA Method 3520C Rev 3, and EPA Reg 8 SOP 508. Analytical methods were EPA method 8015D, Rev 4, and EPA Reg 8 SOP 508, Rev 3.

Semivolatiles via EPA Method 8270: Associated File: GCMS 8270 Semivolatiles W.O. 1001002 and 1001003.pdf,

Volatiles via EPA Method 8260: Associated File: GCMS 8260 Volatiles W.O. 1001003.pdf

TPH/GRO: Associated File: GRO W.O. 1001003 and 1001005.pdf. The amended report (amendment 2) indicates that 34 water samples under WO 1001003 and 10 soil samples (9 under WO 1001003 and 1 under WO 1001005) were received.

QA/QC Criteria for Analytical Methods: QAPP specified and Laboratory specific QA/QC criteria and limits were used as the basis of this ADQ. Note however, the Pavillion QAPP did not provide specific QA/QC criteria associated with the EPA Region VIII Laboratory methods. This QAPP indicates the organic analyses conducted by EPA Region 8 Laboratory are not included other than by reference to the analysis. As such, only corrective actions specified in the EPA Region 8 OPs can evaluated. The laboratory did provide a QA/QC Summary table (attached as a pdf file entitled R8 Lab Summary QA_QC.pdf). The DoD LCS study refers to a study used to derive statistical control limits for Semivolatile and Volatile analytes in laboratory control samples (spiked blank matrix). The QA/QC Summary table, DoD statistical limits, and information gathered during a TSA at Region VIII (unrelated to this project) were used to evaluate the laboratory against data quality indicators and to assess the usability during this ADQ. Table 1 below is a summary of these QA/QC criteria. No information was provided on field QA/QC sample identification, as such none of the data could be validated against field QA/QC samples.

Table 1. Region VIII Laboratory QA/QC Requirements.

QC Type	Semivolatiles (Method 8270D)	DRO (Method 8015D)	GRO (Method 8015D)	Volatiles (Method 8260C)	Frequency
Method Blanks	Preparation Blanks (same as method blank), one with each set of extraction groups within the lab, calibration blanks, <RL	Preparation Blanks (same as method blank), <RL	Instrument Blank (IBL) is the method blank <RL	Method Blank <RL	One per sample set
Surrogate Spikes	"System Monitoring Compounds" use DoD derived limits. concentration 5 ug/mL (20 for tribromophenol) with no dilution. Note, for the Pavillion specific compounds, the surrogate 2-fluorophenol limit is 60-120% in the associated laboratory reports.	60-140% of expected value, o-terphenyl	70-130% of expected value, bromofluorobenzen, added automatically by autosampler	Statistical Limits from DoD LCS Study	Every field and QC sample
Internal Standards Verification.	Every sample, EICP area within ±50% of last ICV or first CCV. Add additional IS if needed for dilutions.	NA	NA	EICP area within -50% to +100% of ICAL midpoint standard	Every field and QC sample for applicable methods

	(SOP Sections 9.4 and 11.4.6)				
Initial multilevel calibration	ICAL: minimum of 6 levels (.25 -12.5 ug/L) , one is at the MRL (0.50 ug/L), prior to sample analysis (not daily) RSD≤20%, r^2 ≥0.990	ICAL: 10-500 ug/L RSD<=20% pr r^2>=0.990	ICAL: .25-12.5 ug/L for gasoline (different range for other compounds) RSD<=20% pr r^2>=0.990	ICAL, RSD<=20% pr r^2>=0.990	As required (not daily if pass ICV)
Initial and Continuing Calibration Checks	CCV (same source as ICAL): daily and every 12 hours, 80-120% of expected value	Daily with each sequence. ICV1 =DRO, ICV2 = surrogate only check 80-120% of expected value	Daily with each sequence. ICV1 & CCV1 = gasoline, ICV2 & CCV2 = BTEX+MTBE+naphthalene 80-120% of expected value	ICV (second source) % R ±20% CCV % R ±20%	CCV At beginning of sample set, every tenth sample, and end of sample set
Second Source Standards	ICV1 is from a second source (includes 7 special compounds) Once after each ICAL, 70-130% of expected value	ICV1 is from a second source, 80-120% of expected value	ICVs are from different source. 80-120% of expected value	ICV (second source) % R ±20%	Each time calibration performed
Standard Reference Material (SRM)	Once per batch, limits based on SRM certification	See below	See below	NA	
Laboratory Control Samples (LCS)	Blank Spike, one per extraction group included once per sequence or every 20 samples. 1mL into 1 L of sample at mid level. Statistical Limits	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value.	Spike Recovery within Statistical Limits from DoD LCS Study	One per analytical batch or every 20 samples, whichever is greater

	from DoD LCS Study (rounded to 0 or 5)				
Matrix Spikes (MS)	Same as LCS	Same as LCS (70-130%, may develop statistical based in future)	Spike with ICAL mix Gasoline 70-130%, others DoD limits	Spike Recovery within Statistical Limits from DoD LCS Study	One per sample set or every 20 samples, whichever is more frequent
MS/MSD	Once per batch or every 20 samples. RPD ≤ 20% Note, the limits in the Reg VIII lab files is ≤ 30%	RPD ≤ 25	RPD ≤ 25	RPD≤30%	One per sample set or every 20 samples, whichever is more frequent
Detection Limit Standard (CRL)	run MDL study approximately annually	DL= RL, ICAL run down to 10 ug/L MDL study approximately annually	DL= RL, MDL study approximately annually	±50% of expected value	CRLs not routinely analyzed, only report to RL (lowest standard of cal model)
Reporting Limits*	0.1 µg/L (generally)[1]	20 µg/L[1]	Gasoline is 20 µg/L[2] Other compounds RL is from 1-200, compound specific	Not specified in QAPP, as EPA RSK was doing the analysis for Killdeer	NA (part of ICAL)
Other Method Specific	GC/MS tuning (DFTPP) : prior to ICAL and at beginning of each 12-hour period.			GC/MS tuning (BFB): prior to ICAL and at beginning of each 12-hour period.	

[1]Based on 1000 mL sample to 1 mL extract

[2]Based on a 5 mL purge

*these limits are compound dependent

2. Summary of Assessment

Note, the terms Findings and Observations are based upon the definitions in the SOP LSAS-QA-02-0, Performing Audits of Data Quality

OBSERVATIONS

1. **Recalculations do not match reported.** The values for two 8260 VOC compounds (Adamantane and 1,2-dimethyl adamantane) varied slightly from the reported (within 10%), see Question 10 below.

2. **Corrective Actions for QC.** The corrective actions the laboratory takes when QA/QC is not met is not always specified, none are specified in the analytical methods.

3. **Sample Preservation.** There is no clear indication of how the samples are stored (e.g. at 4°C) once they are received by the lab for analysis, see Question 4 below. During the TSA conducted on July 26, 2011 the assessment identified that the laboratory does have a good practice of storing samples in a controlled environment that is monitored. All samples must be logged in and out of the sample storage location and this location is controlled with a logged door, and refrigerators that are also locked.. Though not explicit in the laboratory reports, the evidence from the TSA would indicate samples were adequately stored when not undergoing analysis.

4. **Holding Times.** Holding times were not met for all samples, see Question 8 below. The associated samples were correctly qualified with the exception of the GRO samples that were placed on the autosampler within the holding time, but not analyzed until slightly beyond the 7 days.

5. **Data Quality Indicator QC limits exceeded.** The QC results did not meet all the requirements specified in the Laboratory QA/QC Table or associated Method SOP.
    a. **DRO:** The SRM2 sample (lab ID 1000011-SRM2, sample sequence 1001002-22), spiked at 107 mg/L DRO, associated with the four samples had a 133% recovery, which is outside the specified limits from the laboratory provided QA/QC Summary table. No corrective action is specified in that table. In discussions with the laboratory during the TSA conducted on August 26th, they indicated the SRM is considered a LCS, but the limits used to evaluate the result are those submitted with the SRM certification. The sample report (page 139 of 481, sample sequence

number 1281038) also indicates this sample had phthalate contamination (retention time 20.79 minutes) and this is evident on the chromatogram. The laboratory identified this phthalate contamination and was able to review each sample for evidence of phthalates. Sample PGDW5 (lab number 1001002-03) appears to have a similar peak as seen on page 44 of 481, but much lower than observed in SRM2 (relative to the surrogate o-terphenyl). None of the other associated samples appear to have this phthalate. SGDW5 was reported with a DRO concentration at 75.2 mg/L.

i. Method blank spike (1000011-BS1) also have large recovery (443%) and again phthalate contamination was evident as seen on the chromatograph on page 148 of 481 at the same retention time observed in SRM2. Again, this contamination is only observed in sample PGDW5, of the four samples reviewed in this ADQ.

ii. Both SRM2 and the method blank spike were extracted with PGDW5 according to the extraction log on pages 22-23 of 481. Sample PGDW5 should be qualified, though the concentration of the phthalate contamination is considerably less than observed with the laboratory QC samples. For example, in SRM2 the phthalate peak is approximately 7 times the peak height of the surrogate, with a 133% recovery. The phthalate peak is at least an order of magnitude greater than the surrogate in the method blank spike. In sample SGDW5, the peak at approximately 20.7 minutes is roughly 60% of the surrogate.

iii. Field blank phthalate contamination is discussed in the Blank Contamination Memo from the Region 8 Laboratory. Since the field blank identity was not provided for this ADQ, this cannot be evaluated. However, the DRO overlays were reviewed and the sample SDGW05 duplicate also contains the same peak at a retention time of approximately 20.7 minutes indicating it also should be qualified. It is unclear why the laboratory only qualified sample PGSW02D, unless this is due to the low concentration of the contaminant peak in samples SDGW05 and 05 duplicate relative to both the laboratory QC samples, and sample PGSW02D.

b. **SVOC.** PDGW32 for adamantine and 1,3 dimethyl adamantance. The surrogate nitrobenzene-d5 had a recovery of 136%. Since the recovery was high, only detected compounds associated with this surrogate require qualification. As such, adamantine, and 1.3-dimethyl adamantine should be qualified.

EDITORIAL COMMENTS

1. **DRO Analysis Method.** The results report for DROs lists 8015**B** as the analysis method, see Question 7 below in table. This should be 8015D.

ITEMS REVIEWED

Number	ADQ Issue	Yes	No	NA	Comments
File Information					
1	Provide File names: See Inventory of Document-N&C.doc file, provided upon request.				
Sample Information					
2	Are samples uniquely identified and correctly transcribed throughout the data package to the summary of results?	**X**			Samples are uniquely labeled as PGDW5, PGDW20, PGDW30, and PGDW32 for all methods. In addition, samples are identified by unique Lab IDs throughout the raw data packages for all methods.
3	Does sample collection documentation indicate that samples were collected as described in the QAPP, and the schedule and volumes in the planning documentation?		**X**		The only sample collection documentation within the report files is: date/time sample was collected, sample volume and pH for DROs and number of samples collected. Any additional specific sampling information is not expected to be in the laboratory report. So, this is acceptable.
4	Does sample collection documentation indicate appropriate preservation?	**X partial**			According to the Pavillion QAPP, none of the samples for Reg VIII were to be acidified in the field. DRO samples were acidified upon receipt at the lab for analysis. All samples were preserved on ice during shipment. There is no clear indication of how the samples were preserved after receipt by the labs (e.g. temperature stored at).
5	If applicable, is chain-of-custody documentation	**X**			COC documentation was found in files associated with specific work orders/batches. See Excel spreadsheets for further details